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(54) (Title of the Invention) MANUFACTURING METHOD OF WATER ABSORBENT  
CROSSLINKED RESIN

(57) Abstract

(Constitution) A manufacturing method of a water absorbent crosslinked resin characterized by the fact that after forming a resin composition containing 10 to 100 pts. wt. of (b) a hydrogenated diene-based copolymer to 100 pts. wt. of (a) an olefinic thermoplastic elastomer made from 15 to 55 wt.% of a polyethylene-based polymer and 85 to 45 wt.% of an ethylene propylene diene polymer, and containing to 100 pts. wt. the aforementioned component (a) + (b), 5 to 95 pts. wt. of (c) a water absorbent resin, 1 to 25 wt.% of (d) an inorganic filler, 2 to 50 pts. wt. of (e) a softening agent, and 2 to 50 wt. pts. of (f) a plasticizer, the result is irradiated with ionizing radiation.

(Effects) The present invention has superior mechanical physical properties, form retention, and the like, and is suited for various waterproofing seals, water stopping agents, and the like.

(Scope of Patent Claims)

(Claim 1) A manufacturing method of a water absorbent crosslinked resin characterized by the fact that after forming a resin composition containing 10 to 100 pts. wt. of (b) a hydrogenated diene-based copolymer to 100 pts. wt. of (a) an olefinic thermoplastic elastomer made from 15 to 55 wt.% of a polyethylene-based polymer and 85 to 45 wt.% of an ethylene propylene diene polymer, and containing to 100 pts. wt. the aforementioned component (a) + (b), 5 to 95 pts. wt. of (c) a water absorbent resin, 1 to 25 wt.% of (d) an inorganic filler, 2 to 50 pts. wt. of (e) a softening agent, and 2 to 50 wt. pts. of (f) a plasticizer, the result is irradiated with ionizing radiation.

(Detailed Explanation of the Invention)

(0001)

(Industrial Application) The present invention relates to a water absorbent resin foam containing a water swelling macromolecular water absorbing agent and an olefin thermoplastic elastomer. In more detail, it relates to a manufacturing method of a water absorbent crosslinked resin in which a water absorbent resin is blended with an olefin thermoplastic elastomer useful in civil engineering such as waterproofing seals between segments in water and sewer construction and tunnels, and seals in gaps in the outer wall panels of buildings, as well as in water cutoff material in construction work, condensation prevention material with anti-seismic functions, and the like.

(0002)

(Prior Art and Problems) Conventionally, water swelling compositions in which crosslinked polyacrylate, starch-polyacrylate-based resin, vinyl alcohol copolymer resin, resin in which the reactive product of maleic anhydride copolymers and alkaline substances is crosslinked, and other highly water absorbent resins were blended with diene-based rubber and the product was vulcanized were widely used as a waterproof sealing material between segments in tunnels and water and sewer construction (Japanese Unexamined Patent Application Publication S57-108143, Japanese Unexamined Patent Application Publication S57-135160, and the like). However, such water absorbent resin compositions had problems such as a decrease in productivity and an increase in forming costs due to the need for a vulcanization process.

(0003) Also, water absorbent resin compositions in which thermoplastic resin is caused to contain highly water absorbent resin such as, for example, blends where finely divided water absorbent polymers (low-grade olefin-maleic anhydride crosslinked with urea resin or melamine resin) are blended in polybutadiene or ethylene-vinyl acetate copolymers (Japanese Unexamined Patent Application Publication S55-8424), mixtures where highly water absorbent resin (maleic anhydride-isobutylene copolymer, acrylic acid-vinyl alcohol polymer, and the like) is mixed with a composition of chlorinated polyethylene and polyisobutylene (Japanese Unexamined Patent Application Publication S62-106879) and the like have been proposed, but the former has a workability problem where it is strongly dependent on temperature, and in particular, its water absorbency and plasticity are poor at low temperatures, and the latter has problems in terms of environmental protective measures such as that hydrogen chloride is produced during incineration.

(0004) Proposals have also been made such as to enhance the form retention after water absorption by melt-kneading a water absorbent resin containing carboxylate and an olefin containing a glycidyl group to make a polyolefinic resin composition, or to melt-knead water absorbent resin powder, polyolefinic resin pellets and olefinic resin pellet powder (Japanese Unexamined Patent Application Publication S63-110226, Japanese Unexamined Patent Application Publication S63-135431), but these are complex compositions with a thermoplastic polymer base, and have durability problems with prolonged use such as that the water absorbency efficiency drops and that the so-called gel-loss decrease [*sic*]<sup>1</sup> occurs when the water absorbent agency is gradually lost from the resin composition after repeated water absorption and drying.

(0005) The present inventors have investigated non-vulcanized-type water absorbent resin compositions with an olefin thermoplastic elastomer, and have discovered that (1) resin compositions in which water absorbent resin, a softener and a plasticizer are blended with an olefin thermoplastic elastomer made from polyethylene-based polymer and ethylene propylene diene copolymer have superior plasticity and formability, (2) in compositions where inorganic filler is blended with any of the aforementioned components, the dispersion of water absorbent resin in the composition is favorable and such

<sup>1</sup> [Translator's note: *Decrease* appears to possibly be a misspelling for *phenomenon*.]

compositions have superior water absorbency, (3) foaming the aforementioned water absorbent resin composition to a low diameter results in more superior water absorbency, (4) irradiating the aforementioned low diameter foamed product with ionizing radiation increases the strength and increases the weight retention, and (5) blending hydrogenated diene-based copolymer with any of the aforementioned components increases the water absorbency even without foaming and is superior in plasticity, water absorbency and formability, and have filed patents therefor.

(0006) These water absorbent resin compositions have superior water absorbency, plasticity and the like, but an increase in the durability such as preventing water absorbency resin loss (gel loss), such as to increase the performance even further is desired, accompanying an expansion in usage and in the use quantity. Accordingly, the problem of the present invention is to provide a manufacturing method of a water absorbent crosslinked resin with a olefin thermoplastic elastomer as a base having superior mechanical properties, etc., and superior in plasticity and productivity without changing form due to gel loss in the resin composition after water absorption.

(0007)

(Means for solving the problem) To solve the aforementioned problem, the present inventors irradiated the formation of the aforementioned water absorbent resin composition, and verified that the resulting crosslinked construction solved the aforementioned problem, arriving at the present invention as a result of repeated dedicated investigations. In further detail, the present invention provides a manufacturing method of a water absorbent crosslinked resin characterized by the fact that after forming a resin composition containing 10 to 100 pts. wt. of (b) a hydrogenated diene-based copolymer to 100 pts. wt. of (a) an olefinic thermoplastic elastomer made from 15 to 55 wt.% of a polyethylene-based polymer and 85 to 45 wt.% of an ethylene propylene diene polymer, and containing to 100 pts. wt. the aforementioned component (a) + (b), 5 to 95 pts. wt. of (c) a water absorbent resin, 1 to 25 wt.% of (d) an inorganic filler, 2 to 50 pts. wt. of (e) a softening agent, and 2 to 50 wt. pts. of (f) a plasticizer, the result is irradiated with ionizing radiation. The manufacturing method of the water absorbent crosslinked resin of the present invention is described below.

(0008) An olefin thermoplastic elastomer, (a) the base of the water absorbent crosslinked resin of the present invention, is a composition made from a polyethylene-based copolymer (a first component) and an ethylene propylene diene copolymer (a second component). Here, an ethylene homopolymer; ethylene and propylene or another  $\alpha$ -olefin copolymer; a copolymerization of ethylene, vinyl acetate and ethyl acrylate or the like; or homopolymers thereof; or a blend of a homopolymer and copolymer; or the like may be used as the polyethylene-based polymer for the first component. In concrete terms, polyethylene or ethylene-vinyl acetate copolymer is preferred.

(0009) A melt index (MI, JISK 7210, load of 2.16 kg) of 0.01 to 100 g/10 minutes and a density of 0.910 to 0.935 g/cm<sup>3</sup> are preferable for the polyethylene. The polyethylene includes  $\alpha$ -olefin with around 4 to 20 carbon atoms copolymerized to around 20 moles or less. Also, a copolymer with a vinyl acetate content of 10 to 30 wt.% is favorable for the ethylene-vinyl acetate copolymer (EVA), and in particular, one with a vinyl acetate content within a range of 17 to 30 wt.% is favorable. A melt index (190 °C, load of 2.16 kg) of 15 to 25 g/10 minutes is favorable for such an EVA. Ethylene-vinyl acetate copolymer (EVA) is even more favorable as the aforementioned polyethylene-based polymer.

(0010) The ethylene propylene diene monomer (EPDM), the second component of olefin thermoplastic elastomer (a), is a copolymer made from ethylene, propylene and diene.

(0011) Ethylidene norbornene, 1, 4-hexadiene, dicyclopentadiene and the like may be mentioned for the aforementioned diene compound. For the aforementioned EPDM, one whose ethylene content is 50 to 60 mol%, whose propylene content is 20 to 30 mol%, whose diene compound content is 1 to 20 mol% and whose iodine number is 1 to 30 is favorable. For the Mooney viscosity (ML<sub>1+4</sub>, 100°C) of the EPDM, 20 to 100 is favorable, and 50 to 90 is particularly favorable.

(0012) For the blend ratio of the aforementioned polyethylene-based polymer and the ethylene propylene diene monomer (EPDM), the polyethylene-based polymer is 15 to 55 wt.% and preferably 20 to 50 wt.%, and the EPDM is 85 to 45 wt.% and preferably 80 to 50 wt.%. If the polyethylene-based polymer is less than 15 wt.%, the mechanical strength and the formability drop; and if the polyethylene-based polymer exceeds 55 wt.%, the hardness rises, and the tear strength and the like drop.

(0013) The olefin thermoplastic elastomer (a) is manufactured by melt-kneading polyethylene-based polymer and EPDM at the aforementioned ratio. In further detail, 15 to 55 wt.% of polyethylene-based polymer and 85 to 45 wt.% of EPDM are kneaded by a kneader such as a biaxial extruder, monoaxial extruder, roll kneader, Banbury mixer, Brabender or the like. The kneading temperature may be arbitrarily set at a temperature at or above that where the resin component that is the base melts, but ordinarily, a range of 90 up to 160 °C is favorable. For the melt index (190 °C, load of 2.16 kg) of the olefin thermoplastic elastomer composition thus obtained, 0.5 to 20 g/10 minutes is favorable.

(0014) According to the present invention, for the olefin thermoplastic elastomer (a), a hydrogenated diene-based copolymer (b), a water absorbent resin component (c), an inorganic filler (d), a softener (e) and a plasticizer (f) described below may be added at one time or in an arbitrary sequence and kneaded in order to prepare a component independent from the polyethylene-based polymer and the EPDM.

According to the present invention, (b) the hydrogenated diene-based copolymer may be blended in to improve the water absorbency of the water absorbent crosslinked resin.

(0015) The hydrogenated diene-based copolymer is an (A) – (B) block copolymer made from a vinyl aromatic compound polymer block (A), a conjugated diene-based polymer or vinyl aromatic compound, and a conjugated diene and random copolymer block (B); an (A) – (B) – (C) block copolymer made from a taper block (C) in which those vinyl aromatic compounds of the vinyl aromatic compounds and conjugated diene increase gradually; or an (A) – (B) – (A) block copolymer made from the vinyl aromatic compound (A); this is hydrogenated, at least 80% of the conjugated diene portion of the double bonds are saturated, and the number average molecular weight shows 50,000 to 600,000. The above is disclosed in Japanese Unexamined Patent Application Publication H3-72512. For the aforementioned hydrogenated diene-based copolymer, bound styrene of 10 to 30 wt.%, and a melt flow rate (230 °C, load of 2.16 kg) of 3 to 6 g/10 minutes is favorable.

(0016) A commercially available highly water absorbent resin may be used without any particular limitation as the absorbent resin (c) in the water absorbent crosslinked resin of the present invention, but using a highly water absorbent resin obtained by using a crosslinking agent to crosslink a polymer containing an  $\alpha$ , or  $\beta$ -unsaturated compound containing 1 or 2 carboxyl bases or bases which can be derived from carboxyl bases in its molecule as a monomer component is preferable, and the grain diameter is 5 to 30  $\mu\text{m}$ , preferably 10 to 20  $\mu\text{m}$ .

(0017) As examples for such a water absorbent resin, polyacrylate-based resin, starch-acrylate graft polymer, vinyl acetate copolymer, maleic anhydride copolymer, vinyl alcohol copolymer and the like may be mentioned; among these, a polyacrylate-based resin may be used preferably. It is desirable that the aforementioned water absorbent resin have a water absorption capacity such that the water absorbency is 10 to 500 times its own weight, and preferably 50 to 200 times thereof.

(0018) By using an inorganic filler (d) in the water absorbent crosslinking resin of the present invention, the dispersion of the water absorbent resin in the composition improves, so a compatibilizer, in particular, need not be used. Talc, calcium carbonate, gypsum, carbon black, clay, kaolin, silica, diatom earth, magnesium carbonate, barium carbonate, magnesium sulfate, barium sulfate, calcium sulfate, calcium phosphate, aluminum hydroxide, zinc oxide, magnesium hydroxide, calcium oxide, magnesium oxide, titanium oxide, alumina, mica, glass microspheres, zeolite, clay silicate, cement, silica fume, mica powder, and the like may be used for the inorganic filler (d); of these, talc, calcium carbonate and silica are favorable. A powder form, globular form, flake form or other form may be used for these inorganic fillers.

(0019) To increase the plasticity at low temperature, increase the powder adhesion, and improve the workability of the water absorbent crosslinked resin of the present invention, a softener (e) and a plasticizer (f) are blended in. A mineral oil softener may be used favorably for the softener (e). Paraffin, naphthene, aromatic series and other petroleum softeners, a polymerized strong aromatic oil with a high boiling point, liquid paraffin, white oil and the like may be mentioned as mineral softeners, though preferably a petroleum softener, and in particular EPDM, and other good paraffin petroleum softeners with compatibilization may be used favorably.

(0020) A substance used in ordinary resin compositions may be used for the plasticizer (f); for example, a phthalic acid derivative may be used favorably. Dimethyl phthalate, di (2-ethylhexyl) phthalate, diethyl phthalate, dibutyl phthalate, di-n-octyl phthalate, diisooctyl phthalate, diisobutyl phthalate, diheptyl phthalate, diphenyl phthalate, di-isodecyl phthalate, di-tridecyl phthalate, diundecyl phthalate, benzyl phthalate, butyl benzyl phthalate, dinonyl phthalate, alkyl benzyl phthalate, dimethoxy ethyl phthalate, dimethyl cyclohexyl phthalate, methyl phthalyl ethyl glycolate, ethyl phthalyl ethyl glycolate, butyl phthalyl butyl glycolate, and the like and mixtures thereof may be mentioned as phthalic acid derivatives. Besides the aforementioned components, an ultraviolet ray absorbing agent, a thermostabilizer, a light

stabilizer, a flame retarder, an antistatic agent, a nucleating agent, an coloring agent and the like may be further arbitrarily blended in as desired for the present invention.

(0021) For the blending ratio of the aforementioned olefin thermoplastic elastomer (a), a hydrogenated diene-based copolymer (b), a water absorbent resin component (c), an inorganic filler (d), a softener (e) and a plasticizer (f) in the water absorbent crosslinking resin of the present invention, to 100 wt. pts. of component (a), 10 to 100 wt. pts. of component (b), and to 100 wt. pts. of the aforementioned component (a) + (b), 5 to 95 wt. pts. of component (c), favorably 30 to 60 wt. pts. thereof, 1 to 25 wt. pts. of component (d), favorably 3 to 15 wt. parts thereof, 2 to 50 wt. pts. of component (e), favorably 30 to 45 wt. pts. thereof, and 2 to 50 wt. pts. of component (f) are blended.

(0022) If the component (b) is less than 10 wt. pts., the improvement in the water absorbency of the composition will be inadequate, and if it exceeds 100 wt. pts., the weight retention will drop. If the ratio of the water absorbent resin (c) is less than 5 wt. pts., the water absorbency of the composition will be inadequate, and if it exceeds 95 wt. pts., the mechanical strength of the composition will drop. If the ratio of the inorganic filler (d) is less than 1 wt. pt., the dispersion of the water absorbent resin will drop, causing inadequate water absorbency, and if the ratio exceeds 25 wt. pts., the plasticization will drop, and the loss of water absorbent resin will increase. If the ratio of the softener (e) is less than 2 wt. pts., the plasticity will be inadequate, and if it exceeds 50 wt. pts., the formability will worsen. Also, if the ratio of the plasticizer (f) exceeds 50 wt. pts, the kneading operation will be difficult.

(0023) In order to obtain the water absorbent crosslinking resin of the present invention, first, melt-knead each of olefin thermoplastic elastomer (a), a hydrogenated diene-based copolymer (b), a water absorbent resin component (c), an inorganic filler (d), a softener (e) and a plasticizer (f) at the aforementioned ratios in an arbitrary sequence. In order to improve the uniform dispersion of the water absorbent resin in the composition, however, it is favorable to use a Henschel mixer or other such agitator to mix in advance the water absorbent resin (b) and the olefin thermoplastic elastomer component (a) prepared beforehand from polyethylene-based polymer and EPDM. After adding components (c), (d) and (e) to this reserve mixture, melt-knead the product using a kneader such as a monoaxial extruder, biaxial extruder, Banbury mixer, knead roll, Brabender or kneader ruder. Here, the kneading temperature may be arbitrarily set in a range higher than the temperature at which the resin component which is the base melts, but ordinarily, a range of 90 to 250 °C is favorable. The kneaded product obtained is next formed into a sheet or other desired form.

(0024) For the water absorbent crosslinked resin according to the present invention, ionizing radiation is radiated and a crosslinked construction formed to prevent gel loss and improve the durability. Depending on the irradiation conditions (radiation quantity, etc.) of the ionizing radiation, it is possible to retain a crosslinked construction in the entire water absorbent crosslinked resin, or hold a crosslinked construction on both surface layers or one surface layer of the formed product. Through this crosslinking process, the mechanical strength and hot water resistance are improved, and gel loss is suppressed. In particular, formed products with a crosslinked construction made with a high degree of crosslinking only on the surface layer have an improved mechanical strength due to the surface crosslinked layer, and it is also possible to improve the loss prevention of water absorbent resin, form retention, hot water resistance and the like when water absorption and drying are repeated.

(0025)  $\alpha$ -rays,  $\beta$ -rays,  $\gamma$ -rays and X-rays may be mentioned as ionizing radiation to irradiate in order to cause a crosslinking structure. A favorable amount of radiation is in the range of 0.1 to 20 Mrad. If 0.1 Mrad or below, the crosslinking will be inadequate, and if 20 Mrad or higher, the degree of crosslinking will be too high and the water absorbency will drop, and deterioration of the form will occur, so that is unfavorable. The acceleration voltage and quantity of the radiation to be irradiated is arbitrarily set according to the degree of crosslinking aimed for. For example, it is possible to provide effects similar to those of laminating the surface layer with a film in order to prevent gel loss by using a comparatively low acceleration voltage or radiation quantity and causing moderate crosslinking in the surface layer. Also, by using a comparatively high accelerated voltage, the entire crosslinking may be made uniformly crosslinked, and the balance of water absorbency, strength, gel loss prevention performance and the like made favorable. The accelerated voltage and quantity of the radiation to be radiated needs to be altered according to whether the entire formed product is to be crosslinked, whether only the surface is to be crosslinked, etc., and the degree of crosslinked that is aimed for; however, a range of 100 to 850 KV is favorable, and a range of 1 to 8 Mrad is favorable for the amount of radiation.

(0026)

(Embodiments) The present invention is further described in detail using embodiments and a comparative example, but the present invention is not limited to the below. In the embodiments and the comparative

example, the below items were used as resin material and additives. After making a reserve mixture using a Henschel mixer at room temperature with the ratio of olefin thermoplastic elastomer: EPDM (propylene content of 28 wt.%, iodine number of 15, Mooney viscosity (ML<sub>1+4</sub>, 100 °C) of 88) and EVA (vinyl acetate content of 28 wt.%, melt index (190 °C, load of 2.16 kg) of 20 g/10 minutes) shown in Table 1, roll kneading was carried out on the product at approximately 120 °C, and the product was made into pellets.

(0027) Hydrogenated diene copolymer: Dainaron 1320P (trademarked name) (made by Japan Synthetic Rubber Co., Ltd.[)], bound styrene of 10%, melt flow rate (230 °C, load of 2.16 kg), of 3.5 g/10 minutes, Shore-A hardness of 39, hydrogenation of 99% or higher.

Water absorbent resin: acrylic CS-6S (made by Nippon Shokubai Co., Ltd.; polyacrylate-based).

Inorganic filler: talc (made by Hayashi Kasei Co., Ltd.; micron white 5000 A, average grain diameter of 6 μm).

Softener: Mineral oil (made by Idemitsu Petrochemical Co., Ltd.; PW-380 (trademarked name), paraffin mineral oil).

Plasticizer: DIDP (diisodecyl phthalate).

(0028) Embodiments 1 to 3 and the Comparative Example

After kneading in advance water absorbent resin and olefin thermoplastic elastomer made from EPDM and polyethylene-based polymer in the ratios shown in Table 1 at room temperature using a Henschel mixer, the hydrogenated diene copolymer, inorganic filler, softener and plasticizer were melt-kneaded at 140 °C using a Brabender mixer, the product was extruded from the die, and a sheet formed. Next, this was irradiated with a beam of electrons with the conditions shown in Table 1, making the entire layer into a crosslinked structure, and a water absorbent crosslinked resin was obtained having the physical properties shown in Table 1. For the comparative example, the results are shown without radiating a beam of electrons.

(0029) The method for measuring the physical properties shown in Table 1 are as follows.

(1) Hardness: Measured according to Shore C.

(2) Water absorbency after 1 day and 7 days: The value obtained by dividing the weight of the resin foam after absorbing water by the weight of the resin foam before absorbing water.

(3) Rupture strength: Measured using ASTM D-638 (23 °C).

(4) Weight retention: The value obtained by dividing the weight obtained by weighing after swelling and then drying once by the weight before swelling (the smaller this value is, the higher the gel loss from the water absorbing agent).

(5) Hot water resistance: The state after the composition shown in Table 1 was immersed in 70 °C water for 72 hours was evaluated using the below standards.

○: Swelling in the composition is uniform in three directions and the surface is smooth

×: Swelling in the composition is not uniform in three directions, curls appear in the crosslinking, and the surface is markedly rough

(0030) As is made clear in Table 1, the water absorbent crosslinking resin according to the present invention has a favorable rupture strength, weight retention and hot water resistance. In contrast to this, it can be seen that the water absorbent crosslinked resin not irradiated with a beam of electrons has a strength and hot water resistance that is inferior.

(0031)

(Table 1)

Table 1

		Embodiment			Comparative Example
		1	2	3	1
*	EVA	25	25	25	25
	EPDM	65	50	50	50
	Hydrogenated diene copolymer	10	25	25	25
	Water absorbent resin	50	50	50	50
	Softener	20	20	20	20
	Inorganic filler	5	5	5	5

	Plasticizer		20	20	20	20
Electron beam irradiation conditions	Voltage (KV)		800	800	800	-
		Amount (Mrad)	6	6	8	-
Physical properties	Water absorbency	After 1 day	1.1	1.1	1.1	1.1
		After 7 days	5.3	10.0	7.0	11.0
	Rupture strength		18.6	19.0	20.0	9.8
	Weight retention		98	97	98	95
	Hot water resistance		o	o	o	x

\* Wt. pt.

(0032)

(Effects of the Invention) The water absorbent crosslinking resin according to the present invention is made by using olefin thermoplastic elastomer as a base, and forming a composition in which specific quantities of water absorbent resin, inorganic filler, softener and plasticizer are blended, after which the product is crosslinked using ionizing radiation; because the composition before crosslinking is thermoplastic, it has characteristics such as that it can readily be formed into an arbitrary form using sheet forming, contour extrusion forming or the like; it is superior in plasticity and the like so its workability at low temperatures is favorable; it is superior in water absorbency rate and water absorbency due to its hydrogenated diene copolymer blending; it has superior productivity because it can be manufactured without the need for a vulcanizing process, and the like. The water absorbent crosslinked resin of the present invention is crosslinked through ionizing radiation, so it has superior mechanical physical properties, and gel loss is prevented so it has superior form retention. A crosslinked substance obtained using the method of the present invention in this manner can be used in civil engineering such as waterproofing seals between segments in water and sewer construction and tunnels, and seals in gaps in the outer wall panels of buildings, as well as in water cutoff material in construction work, condensation prevention material with anti-seismic functions, and further can be used as a water retention material or the like for agriculture, forestry and gardening.

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